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In re application of:

Donald D. MONTGOMERY

Application Serial No. 09/394,133

Filed: September 10, 1999

For: **ELECTROCHEMICAL SOLID  
PHASE SYNTHESIS**

Art Unit: 1741

Examiner: Smith Hicks, E.

Attorney's Docket No. 16650004US02

**DECLARATION UNDER 37 C.F.R. §1.132**

Commissioner for Patents  
Washington, D.C. 20231

Sir:

This Declaration is submitted in response to the Office Action mailed from the U.S. Patent and Trademark Office on December 10, 1999. An Amendment and Response is submitted herewith.

1. I, Donald D. Montgomery, hereby declare that I am the inventor of the subject matter claimed in the present application.

2. I herein provide some background information to highlight the differences between the materials and methods of the prior art such as those described by Heller *et al.* for *in situ* synthesis and contrast the same with the methods of the present invention.

3. The methods of Heller *et al.* requires using electric fields to transport charged reagents toward and away from selected electrodes. These charged reagents react with appropriate moieties that are immobilized at, or in the vicinity of, the electrode/s to which they are transported. An ancillary aspect of this method is that these immobilized moieties are not protected chemically. In other words, they will always react with the reagents whether the transport mechanism arises from electric fields, convection or diffusion. As a result, the electrode/s where a reaction between the immobilized moieties and the charged reagents is not desired must be 'protected' by applying an electric field that repels the charged reagents. All electrodes must be on simultaneously to either protect or transport the charged reagents. Uncharged reagents cannot be used. Electrochemical processes are not used to create reagents in this method. Further, as set forth, *infra*, electrochemical processes cannot be used to create reagents in this method.

4. The methods of Heller *et al.* may be summarized as follows:

- Uses electric fields to transport charged reagents to reaction sites

- Uses electric fields to transport charged reagents to reaction sites
- Uses electric fields to protect sites where no reaction is desired
- All electrodes in an array must be on simultaneously
- Does not use electrochemistry to create reagents
- Does not generate reagents *in situ*
- No uncharged reagents can be used
- Does not use chemical methods to protect immobilized reactive moieties

5. The methods according to the present invention require using electrochemically generated reagents to cause chemical change proximate to selected electrodes. These reagents and their precursors may be charged or uncharged. Transport of these reagents and their precursors is due solely to diffusion or convection. Electric fields in electrochemical systems are not sufficient to have any material effect on the transport of reagents or their precursors. The sites where a chemical modification is not desired can be protected by chemical methods.

6. The methods of the present invention may be summarized as follows:

- Uses electrochemical processes to generate reagents *in situ*
- Uses uncharged or charged reagents and reagent precursors
- Uses chemical methods to protect immobilized reactive moieties
- Selectively uses electrodes in an array
- Does not use electric fields to transport charged moieties

7. The methods of Heller *et al.* are fundamentally different from the methods according to the present invention. A discussion of some of these differences is set forth following:

- The use electrodes to create electrochemically generated reagents requires a set of physical conditions that are fundamentally incompatible with the use of electrodes to create electric fields to transport charged moieties.

- The method of Heller *et al.* fundamentally excludes the use of uncharged reagents.

- The description of the enabling reduction to practice in the Heller *et al.* patent is not consistent with known scientific facts.

8. Electric fields in electrochemical systems. The transport of molecules in electrochemical systems is completely described by diffusion. The terms in the transport equations that are due to diffusion are five to six orders of magnitude larger than the terms due to either convection or migration. Therefore, both convection and migration have no material effect

on transport in conventional electrochemical systems. Certain specialized electrochemical systems, such as rotating disk and wall jet electrodes, are constructed to increase the magnitude of the convective term such that it becomes material for describing transport in these systems. However, there are no electrochemical systems where the migration term has a material effect.

The physical reason that the migration term has no material effect on transport in electrochemical systems is the electrical double layer that was described by Helmholtz in 1853 (H.L.F. Helmholtz, *Ann. Physik.*, 89:211 (1853)), elucidated in 1910 by Gouy (G.Gouy, *J. Phys. Radium*, 9:457 (1910), G. Gouy, *Compt. Rend.*, 149:654 (1910), D.L. Chapman, *Phil. Mag.*, 25:475 (1913)) and further elaborated by Stern in 1924 (O.Stern, *Z. Elektrochem.*, 30:508 (1924)). The electrical double layer screens the charge on an electrode from the bulk solution. The electric field due to the charge on an electrode is almost completely screened within 5-10 angstrom of the electrode surface.

Likewise, the electric fields that arise from ionic current that is passed during electrochemical processes are not materially important for transport of reagents to electrodes. Migration effects are only observable in regions where there are no concentration gradients. Because electrochemical processes result in changing the chemical identity of a reagent, there are always large concentration gradients that are responsible for transporting molecules to electrodes during electrochemical processes. This is why there are no observable electric field effects on the kinetics or free energy of electrochemical processes such as the reduction of  $\text{Cu}(\text{CN})_4^{4-}$  to Cu and  $(\text{CN})^-$  at electrodes with a negative applied potential. Electric field effects are reduced further in electrochemical systems using electrode array geometries because the current density falls off proportional to  $r^{-2}$ , where  $r$  is the distance from the electrode

The fact that migration has no material effect on transport has important consequences for electrochemical science. For example, electrochemical measurements can be used to determine thermodynamic constants without having to consider electrical work effects. The use of most electrochemical techniques to study reaction kinetics depends on negligible migration effects.

9. Using electrodes to create electric fields. The only method available to construct a system that exhibits electric fields of sufficient magnitude to transport charged moieties is to remove all ancillary ions from the solutions. As a result, the electrical double layer that forms is constructed only from the ions of interest. However, as soon as these ions form an electrical double layer, the electric field disappears. Usually, the electrical double layer is established within milliseconds. These effects are purely capacitive. There are no electrochemical processes involved.

W.A. 10. The examples provided by Heller et al. are not enabling. These examples are not consistent with several well known and material scientific facts including the following:



First, the devices described by Heller *et al.* use electrodes that are composed of aluminum metal. The electrolyte solutions that are described contain chloride ions. It is well known that both aluminum and aluminum oxide dissolves and corrodes very rapidly under these conditions. Further, the corrosion processes are accelerated by orders of magnitude under the current densities that are described in the Heller *et al.* specification. Heller *et al.* allege that they have immobilized a reactive moiety onto the surface of the electrode via covalent linkage to the first monolayer of the thin aluminum oxide layer that covers all aluminum metal surfaces that are exposed to ambient oxygen. Heller *et al.* further allege that these moieties are subsequently modified by charged reagents that are attracted to the electrode by an electric field. Using standard corrosion rates of aluminum oxide in chloride solutions, the first monolayer will be gone within a microsecond. Neither the immobilized reactive moiety nor the aluminum electrode could have survived the conditions described in the Heller *et al.* specification.

Second, in the presence of the electrolyte solutions described in the Heller *et al.* specification, the electrical double layer shields the charge on the electrode from the bulk solution. There should be no transport of charged species after the establishment of an electrical double layer.

11. I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the above-identified application or any patent issuing thereon

Date: June 9, 2000

Donald D. Montgomery, Ph.D.  
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